The Use of an Interference Microscope for Measurement of Extremely Thin Surface Layers

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(Manuscript received March 15, 1956)

A method is given for the thickness measurement of p-type or n-type surface layers on semiconductors. This method requires the use of samples with optically flat and reflecting surfaces. The surface is lapped at a small angle in order to expose the p-n junction. After detecting and marking the p-n junction, the thickness is measured by an interference microscope. Another application of the equipment is the measurement of steps in a surface. The thickness range measurable is from 5×10^{-6} *cm to* 10^{-3} *cm.*

INTRODUCTION

Extremely thin p-type or n-type surface layers can be obtained on semiconductors by recently developed diffusion techniques.[1, 2] Layer thicknesses of the order of 10^{-4} cm are currently used for making diffused base transistors.[3, 4] The thickness of the diffused layer is an important parameter for the evaluation of such transistors. Its measurement is facilitated by lapping a bevel on the original surface, thus exposing the p-n junction within the bevel where the thickness appears in an enlarged scale. With a sharp and well defined angle, one would obtain the thickness by the measurement of the angle and of the distance between the vertex and the p-n junction.

However, it is extremely difficult to obtain vertices sharp enough for measurements of thicknesses of the order of 10^{-4} cm. To avoid this difficulty, an interferometric method was developed in which the depth is measured directly by counting interference fringes of monochromatic light. The method can also be used for the measurement of small steps

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[1] C. S. Fuller, Phys. Rev., **86,** p. 136, 1952.

[2] J. S. Saby and W. C. Dunlap, Jr., Phys. Rev., **90,** p. 630, 1953.

[3] C. A. Lee, B.S.T.J., **35,** p. 23, 1956.

[4] M. Tanenbaum and D. E. Thomas, B.S.T.J., **35,** p. 1, 1956.

and similar problems occurring, for example, in the evaluation of controlled etching and of evaporated layers.

PRINCIPLE[5]

A half-silvered mirror is brought into contact with a reflecting surface. If this combination is illuminated with monochromatic light, one observes interference fringes. Dark lines appear where the distance between mirror and surface is $n \times \lambda/2$, where n is an integer. Between two points on adjacent fringes the difference in this distance is therefore $\lambda/2$. Hence the fringes can be regarded as contour lines for the distance between the mirror and the surface under consideration. Since the mirror is optically flat, one can deduce the profile of the surface. Equidistant and parallel fringes, for example, prove the surface to be flat. By taking the profile across a bevel or a step, one is able to measure the depth of one part of the surface with respect to another optically flat part of the surface. The reflectivity of the crystal surface should be as high as possible, and that of the mirror should be of the same order. The fringes are then produced by the interference of several wave trains which make the fringes very sharp, and one can measure fractions of $\lambda/2$. With the equipment described here, one is able to interpolate to $1/10$ of $\lambda/2$ or less.

Since small linear dimensions are involved, this principle was adapted for use under a microscope. Hence, it is possible to measure small linear dimensions and the correlated depth simultaneously.

The measurement of small steps, or steps not too steep in an otherwise flat surface, can be done without altering the sample. For measurement on steep and high steps a bevel must be lapped on the sample.

For the measurement of p-type or n-type surface layers on semiconductors, it is essential to lap a bevel on the original surface. The p-n junction is thus exposed and can be found by an electrical method. After marking its position within the bevel, it is then possible to measure its depth with respect to the original surface by taking the profile across the bevel. The marking has to be such that it will be visible in the fringe pattern. By a proper adjustment of the optical flat, a fringe pattern can be produced in which the profile is easily interpreted and the depth measurement amounts to a counting of fringes.

PREPARATION OF THE SAMPLES

The method requires the use of samples with optically flat and highly reflecting surfaces with respect to which a depth can be measured. It is

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[5] S. Tolansky, Multiple-Beam Interferometry of Surfaces and Films, Oxford, at at the Clarendon Press, 1948.

also advisable to use plane-parallel samples to facilitate the lapping of a bevel at a small angle.

For lapping. the sample is waxed with its back side to the face of a short steel cylinder. The face is cut at a small angle. Angles of 0.5° or 1.0° are practical. The cylinder is placed in a jig, in such a position that approximately half of the sample surface projects above the plane of the jig (Fig. 1). A short grind on a slightly rough glass plate using a fine abrasive with water gives usable bevels. For a shiny finish just the right degree of roughness of the glass is important. The use of a lapping machine with a vulcanized fiber plate and fine abrasive gives a better surface finish, but the ridge is not as sharp. A 0.5° bevel could be obtained only on a glass plate.

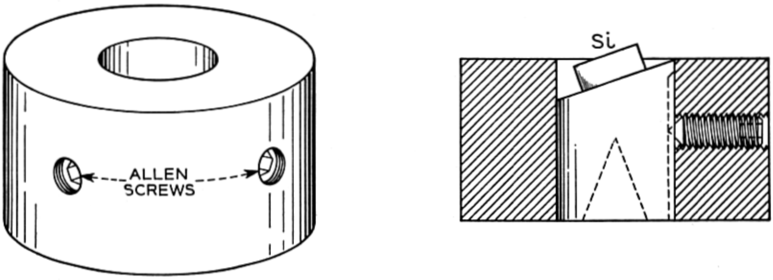

Fig. 1 — Jig for lapping a bevel.

MARKING OF p-TYPE OR n-TYPE SURFACE LAYERS

In a sample with a p-type or n-type surface layer the junction is exposed within the bevel. The next step is to detect and mark the junction.

The sample is fixed on a microscope stage which allows a micrometer controlled movement in two directions (Fig. 2 shows a Wilder micrometer cross slide). The sample is oriented in such a way that the ridge is parallel to one direction of movement (y-direction). One or two lines of aquadag are applied to the surface of the sample, perpendicular to the ridge. The aquadag should be diluted with water in such a proportion as to achieve a thin film which is non reflecting.

A needle is fixed to the base of the stage with a suitable linkage leaving a vertical degree of freedom. The needle is brought into contact with the surface of the sample outside the aquadag. Thus, the sample can be moved under the needle while the needle maintains contact. In a suitable electrical circuit, the needle serves as detector of the junction. The sample is moved in the direction perpendicular to the ridge (x-direction)

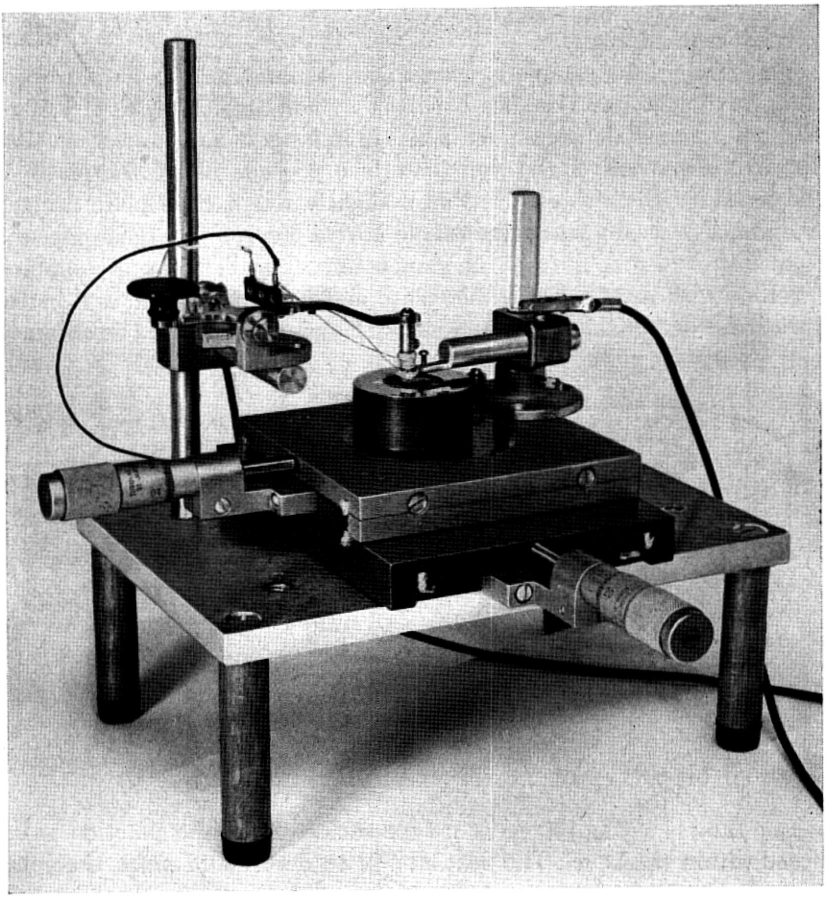

Fig. 2 — Apparatus for locating and marking *p-n* junctions.

until the needle rests on the p-n junction as seen by the electrical detector. By moving the sample in the y-direction the same needle scrapes a line through the aquadag. In this line, the reflecting sample surface is bared and thus, a reflecting line is produced within a non-reflecting surrounding and can be seen in the fringe pattern.

If the ridge of the sample is exactly lined up with the y-direction, the needle moves along the junction and the line in the aquadag indicates the position of the junction exactly. To minimize an error due to poor alignment, it is advisable to locate the junction close to the edge of the aquadag. By doing this on two different sides of the coating, the average of both readings compensates the error. To obtain, however, the maximum of accuracy, one can locate the junction at any point. (See Fig. 3, Point A). Moving the sample in the y-direction scribes a line through the point at which the junction was found. A movement in the x-direction with the needle in the aquadag marks a point B on this line. The distance from this point to the junction can be obtained from the readings on the micrometer. Thus, the exact point at which the junction was located can be reproduced under the microscope.

DETECTION OF THE p-n JUNCTION

1. Thermoelectric Probe

The thermoelectric voltage[6] occurring between a hot and a cold contact to the sample, changes sign by crossing the junction with the hot contact. The advantage of this probe is that it does not depend upon the rectification properties of a p-n junction. The thermoelectric probe is most suitable for germanium since lapping across a p-n junction normally produces a "short" between the two regions. However, it is likely to give a p-reading on lightly doped n-material. It is therefore only usable on heavily doped layers, where the nearly compensated zone is very small. In the case of silicon, the junction normally maintains rectifying properties after lapping; thus, a photocurrent is present. This current is superimposed upon the thermocurrent. Therefore, the thermoelectric probe is only usable in the dark. The photoelectric method (see below) is more convenient for these cases.

The thermoelectric probe used, consisted of a commercial phonograph needle, which had a good hemispherical point and was surrounded by a piece of ceramic tubing carrying a heating coil. Between needle and sam-

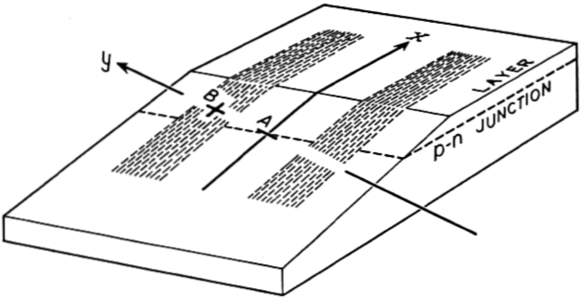

Fig. 3 — Schematic view of a scribed *p-n* junction.

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[6] V. A. Johnson and K. Lark-Horowitz, Phys. Rev., **69**, p. 259, 1946.

ple a sensitive galvanometer is connected. It is unimportant whether the contact is made to the p-type or to the n-type side of the sample. The best results are obtained on freshly lapped and clean surfaces. It is, therefore, advisable to keep the sample on the steel cylinder while applying the thermoelectric probe.

When applying the probe, the sample is moved in the x-direction to the point of zero deflection on the galvanometer, whereby the point rests on the junction.

2. Point Rectification on the Surface

This test is also usable on p-n junctions which are not rectifying. With one fixed ohmic contact to the sample, the point rectification of the movable needle can be displayed on an oscilloscope. By crossing the junction with the needle, the characteristic changes from p-n to n-p. Thus, the needle again can be placed on the junction.

This test was applied on lightly doped Ge-layers. The oscilloscope pattern is not very definite, since on a lapped surface the point rectification is poor. However, with some experience the junction can be located. It is advisable to repeat the measurements several times. Boiling the sample in water before applying the probe improves the surface.

3. Photoelectric Probe

This method requires that the junction exhibit rectifying properties. It is most successfully applied to silicon. Between the needle and a contact to either the p-type side or the n-type side of the sample, a high impedance voltmeter is connected. While the sample is strongly illuminated, it is moved in the x-direction. When the needle crosses the junc-

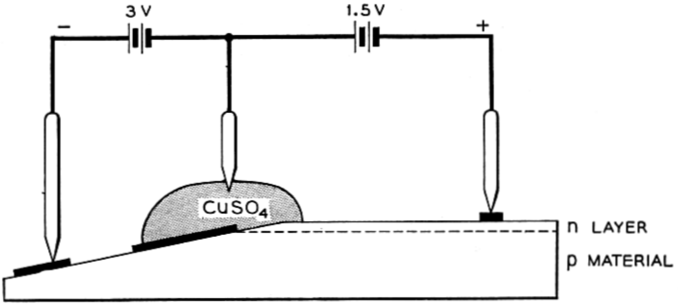

Fig. 4 — Arrangement for Cu-plating the *p*-type side of a *p*-*n* junction.

tion, a change in the photoelectric voltage occurs. For more careful measurements one might plot the photovoltage versus the x-coordinate in units of the micrometer. Such a plot allows an accurate location of the junction in these units. If the micrometer is set for this reading, the needle will rest on the p-n junction.

4. Potential Probe

This is another method for locating the junction where the junction is at least slightly rectifying. One needs two contacts to the sample, one on the p-type side and the other on the n-type side. When a current is passed through the sample in the reverse direction, the voltage between the needle and either contact shows a discontinuity at the junction. The voltage can be plotted in a similar way as described for the previous method, and thus the needle can be set on the p-n junction.

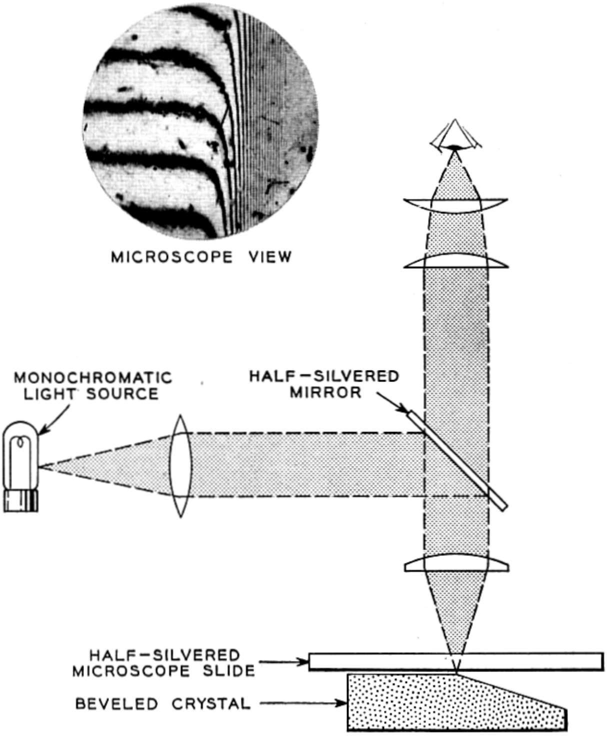

Fig. 5 — Diagrammatic view of the light path in the interferometer.

5. *Plating the p-side of the p-n Junction*[*]

This method detects and marks the junction in one process without using the micrometer arrangement. Voltages are applied in such a way that only the p-type side is plated (See Fig. 4). Since the plating projects up and is not optically flat, it can be recognized under the interferometer. It has the advantage of showing the junction as a line. The disadvantage is that it is only convenient on rectifying p-n junctions (silicon), with n-type layers since the plating ought to take place on the body side of the p-n junction.

THE INTERFEROMETER

The main part of the interferometer is a microscope with illumination through the objective. As a source of monochromatic light, a sodium lamp for which $\lambda = 5.89 \times 10^{-5}$ cm is most convenient. The use of a

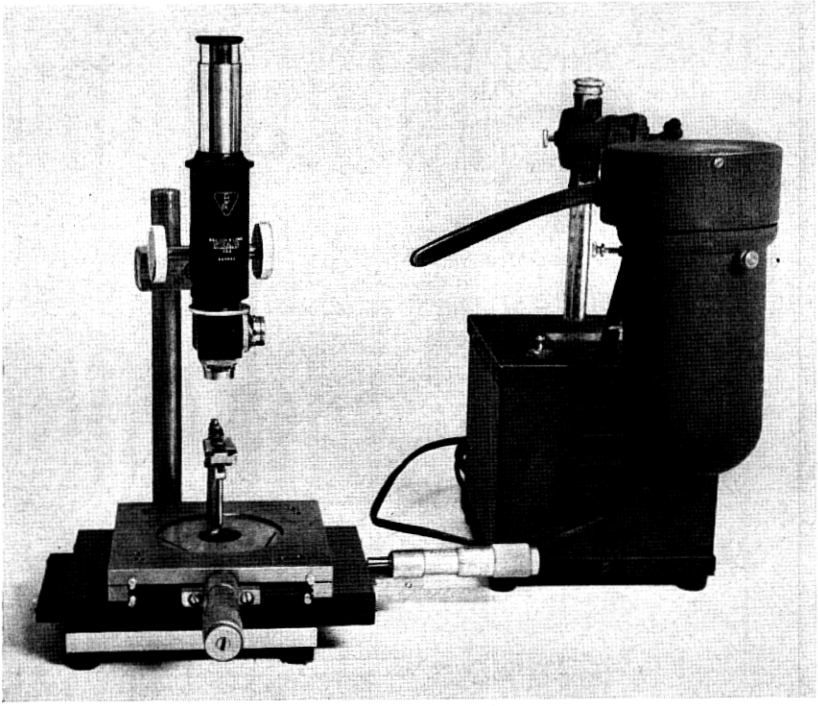

Fig. 6 — Interferometer with light source.

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[*] This method was developed by N. Holonyak.

shorter λ would increase the resolution. However, a sodium lamp gives enough light that one can easily work in daylight.

The microscope is mounted above a micrometer cross-slide of the same kind as used in the procedure for marking the p-n junctions. The stage carries a special sample support. Fig. 5 gives a diagrammatic view of the light path in the interferometer. A normal microscope is used with an attachment carrying a semi-transparent mirror. Fig. 6 shows a photo of the complete arrangement, and Fig. 7 gives the details of the sample support.

The prepared sample is waxed to a microscope slide and covered by a half-silvered mirror. Both are placed on the adjustable lower jaw of the sample support. The lower jaw is raised so that the upper jaw presses against the mirror. In this position it is fixed by tightening the screw in the back. Thus the mirror and sample are in contact, and the fringes can be observed through the microscope. Three screws in the lower jaw make it possible to change the relative position of mirror and sample. Thus the fringe pattern can be adjusted to make it most suitable for the particular case.

THE MEASUREMENTS

The measurement of a layer thickness was chosen to demonstrate the principle of evaluating a fringe pattern. (See Fig. 8.) The first illustration

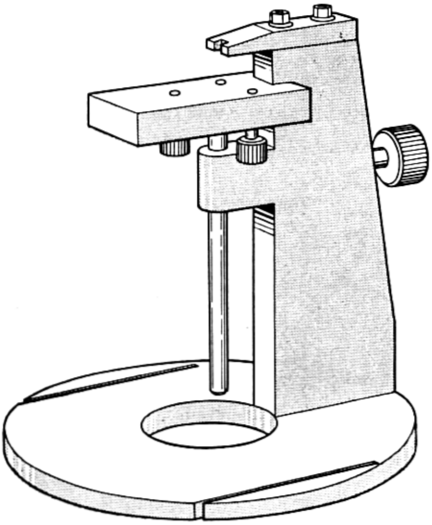

Fig. 7 — Sample support.

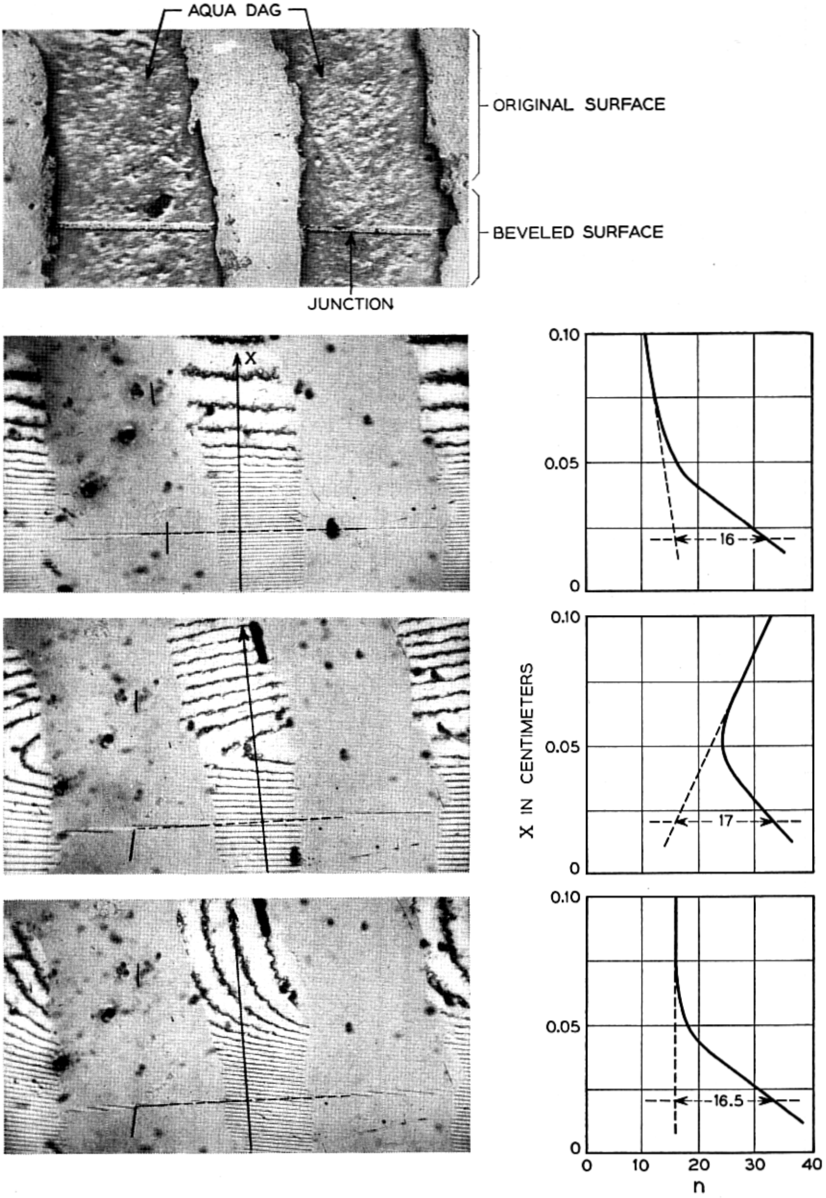

Fig. 8 — Evaluation of the interference fringe pattern on a scribed *p*-*n* junction.

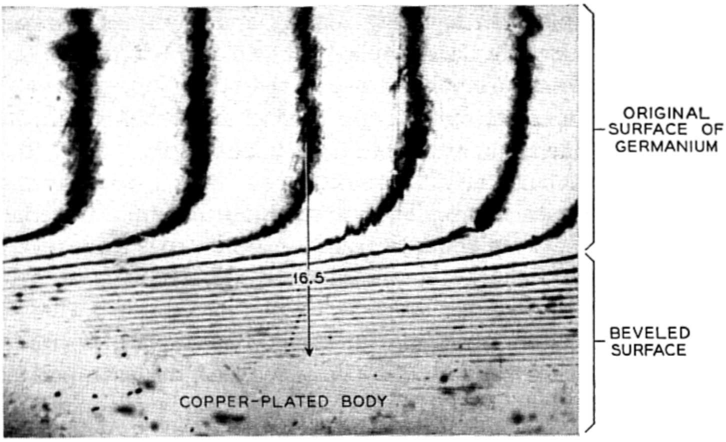

Fig. 9 — Evaluation of the interference fringe pattern on a Cu-plated *p-n* junction.

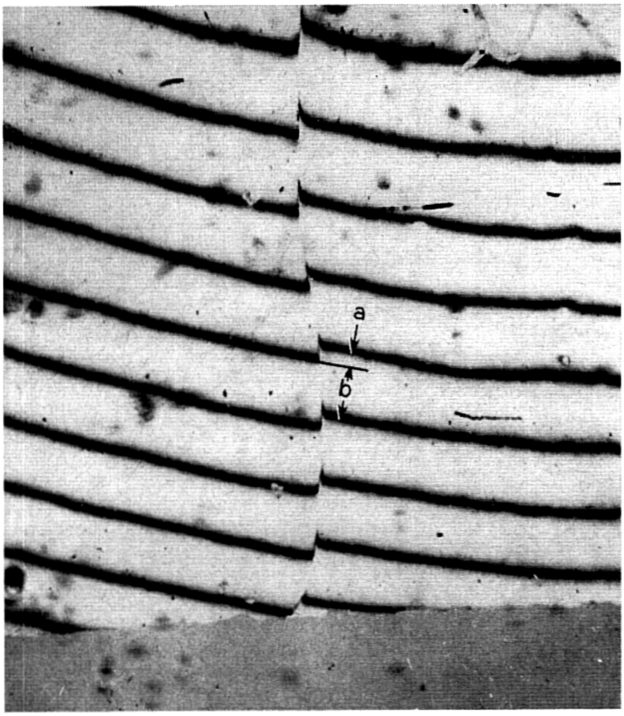

Fig. 10 — Evaluation of the interference fringe pattern of a shallow step in a surface.

shows a sample with aquadag coating and the lines marking the p-n junction. Under this, three typical fringe patterns obtained on this sample are presented. As pointed out in the beginning, one can regard the fringe pattern as contour lines for the distance between mirror and sample. The profile along any arbitrary straight line will show the structure of the sample surface. The profiles along the marked x-axes are shown to the right in each case. They were obtained by plotting the points of intersection between the n-th fringe and the x-axis against n. The original surface and the bevel (in this case 1°) are easily recognized. The dashed line is an extrapolation of the original surface. The vertical line marks the position of the p-n junction. The layer thickness is obtained as the difference in n at this point between the extrapolated original surface and the beveled surface. Note that the beveled surface need not necessarily be flat.

Fig. 11 — Evaluation of the interference fringe pattern of a steep and high step in a surface.

In the second case the fringes turn back. With a slightly different setting of the mirror the fringes could be almost parallel with the turning point outside the field of sight. The fringe pattern then resembles the first case, and therefore it might be easily misinterpreted.

The third case makes the plot unnecessary. The x-axis is chosen in such a way that it coincides with a fringe in the original surface. Thus, in the profile plot, the original surface is horizontal. Hence, the layer thickness can be obtained by counting the number of intersecting fringes between original surface and the p-n junction. This is, therefore, the most convenient setting of the mirror.

The noted number gives in each case the layer thickness in "fringes." All three cases are in essential agreement. The layer thickness in this case is

$$\begin{aligned}\Delta n \times \frac{\lambda}{2} &= (16.5 \pm 0.5) \times \frac{5.89}{2} \times 10^{-5} \text{ cm} \\ &= (4.85 \pm 0.15) \times 10^{-4} \text{ cm}\end{aligned}$$

Fig. 9 gives the fringe pattern obtained with a silicon p-n junction marked by the plating procedure.

The evaluation of steps in a surface is shown for two cases. The very shallow step in Fig. 10 is an example in which fractions of $\lambda/2$ are to be measured. The step here is

$$\frac{a}{a+b} \times \frac{\lambda}{2} = 0.195 \times \frac{5.89}{2} \times 10^{-5} \text{ cm} = 5.75 \times 10^{-6} \text{ cm}$$

In Fig. 11 the step is so high and steep that it is impossible to correlate the fringes crossing the step. But with the aid of the bevel, seen in the lower part of Figure 11, a correlation is possible. The height of the step along the drawn line is

$$(25 - 12) \times \frac{\lambda}{2} = 13 \times \frac{5.89}{2} \times 10^{-5} \text{ cm} = 3.8 \times 10^{-4} \text{ cm}$$

The accuracy of the method depends mainly on the quality of the optically flat mirror since it serves as a plane of reference. A thin mirror is likely to be slightly bent under the pressure of the clamp. Therefore, it is advisable not to work with too high a pressure. For the measurement of layer thicknesses the quality of the original surface is also important. An accuracy of 5 per cent is easily obtained using half-silvered microscope slides for the mirror. These slides are essentially flat over the small region covered by the microscope.